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An American National Standard

Standard Practice for Evaluation of Air Assay Media by the Monodisperse DOP (Dioctyl Phthalate) Smoke Test[1]

This standard is issued under the fixed designation D 2986; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 The dioctyl phthalate (DOP) smoke test is a highly sensitive and reliable technique for measuring the fine particle arresting-efficiency of an air or gas cleaning system or device. It is especially useful for evaluating the efficiency of depth filters, membrane filters, and other particle-collecting devices used in air assay work.

1.2 The technique was developed by the U.S. Government during World War II.[2] Its validity for use in evaluation of air sampling media has been well demonstrated.[3]

1.3 Although a little latitude is permissible in the associated equipment and in the operation method, experience has shown the desirability of operating within established design parameters and recognized test procedures.[4]

1.4 This practice describes the present DOP test method, typical equipment, calibration procedures, and test particles. It is applicable for use with commercially available equipment.

1.5 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Document

2.1 *ASTM Standard:*

D 1356 Terminology Relating to Atmospheric Sampling and Analysis[5]

3. Terminology

3.1 *Definitions:*

3.1.1 *dioctyl phthalate (DOP)*—for the purpose of this method, refers to di-2-ethylhexyl phthalate, technical grade.[6]

3.1.2 *owl*—an optical instrument for visual measurement of the particle diameter of the monodisperse aerosol.

3.1.3 For other definitions of terms used in this method, refer to Terminology D 1356.

4. Summary of Practice

4.1 A monodispersed aerosol of 0.3-μm diameter is continuously generated by condensation of DOP vapor under controlled conditions. By selective value arrangement, a metered portion of this aerosol is drawn through a specimen mount containing the item under test. Flow rate through the specimen is adjustable and the corresponding flow resistance is noted as part of the test.

4.2 With aerosol generation stabilized (constant particle size and concentration), aerosol concentration is measured upstream and downstream of the specimen under test by use of a linear forward light-scattering photometer.

4.3 Results are expressed as percent of DOP penetration at the flow rate used.

5. Apparatus

5.1 Equipment for use with this technique consists of several interoperational parts. These are indicated in proper relative arrangement by the diagrammatic sketch, Fig. 1. In Fig. 1, the letter designations refer to the same parts as described in the immediately following subsections:

5.2 *Air Supply Source* (a)—This can be a blower as shown diagrammatically or a compressed air source with stepdown regulator. In any case, the air supply source must be clean, free of entrainment, and sufficient to provide full flow against the total resistance of aerosol generator and aerosol conductor lines to the excess aerosol exhaust point.

5.3 *DOP Aerosol Generator* (b)—The generator is designed to produce uniform size liquid droplet particles of 0.3-μm diameter at a concentration of about 100 ± 20 μg/L of air. Further description of the generator is given in 7.3.

5.4 *Aging Chamber* (c)—This is simply a large vessel (usually about 20 L in volume) wherein some dwell time is provided to permit stabilization of the aerosol.

5.5 *Sample Holder* (d)—Size and design of the sample holder can be accommodated to the item under test. However, for evaluation of filter media, a circular test area of 100 cm^2 ± 2 % is specified. Provision is made to measure flow resistance across the test piece. A wire screen may be used to support the sample.

5.6 *Particle Size Analyzer* (e)—Particle size in the aerosol is indicated by the particle size analyzer. The visual owl may be used to verify the aerosol particle size. The electronic owl is an adaption designed to remove the human factor; it has proven to be highly successful. Both instruments operate by optical rotation of light scattered at a 90° angle. Paragraph 7.4 gives further detail for the optical owl.

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[1] This practice is under the jurisdiction of ASTM Committee D-22 on Sampling and Analysis of Atmospheres and is the direct responsibility of Subcommittee D22.01 on Quality Control.

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[2] Knudson, H. W., and White, Locke, Ens. USNR, "Development of Smoke Penetration Meters," *Naval Research Laboratory Report No. P-2642*, P.B. No. 119781, September 1945.

[3] Smith, Walter, J., and Surprenant, N. F., "Properties of Various Filtering Media for Atmospheric Dust Sampling," *Proceedings*, ASTM, Vol 53, 1953, pp. 1122–1135.

[4] *Instruction Manual—Penetrometer*, Filter Testing, DOP, Q127 136-300-138B, Edgewood Arsenal, MD, July 1963.

[5] *Annual Book of ASTM Standards*, Vol 11.03.

[6] Available as "Flexor Plasticizer DOP" from Union Carbide Corp., Chemicals Division, New York, NY 10017.

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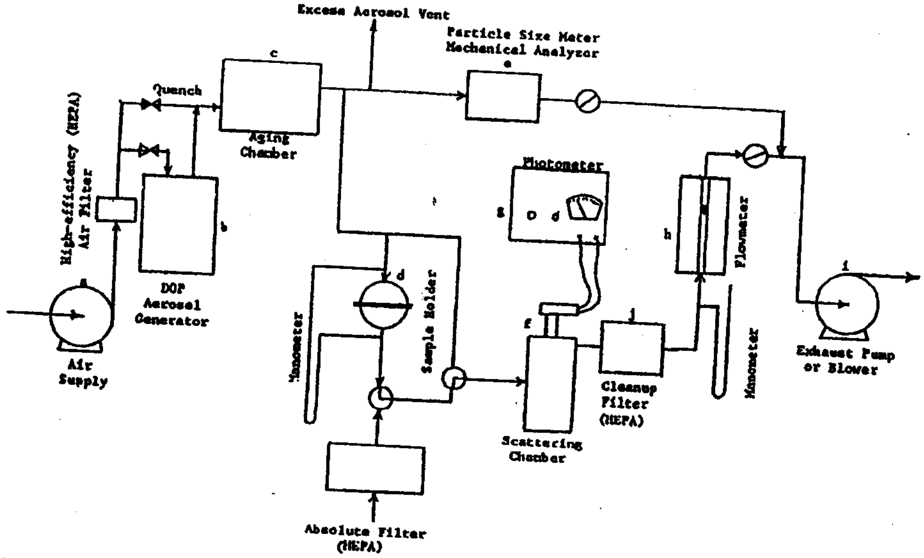

FIG. 1 Simplified Diagram Showing Relation of Principal Parts of DOP Aerosol Test Apparatus

5.7 *Scattering Chamber* (f)—The scattering chamber is used to determine concentration of aerosol either upstream or downstream of the item under test. Further detail for a typical chamber is given in 7.5.

5.8 *Photometer* (g)—This is a combination of sensitive multiplier phototube and meter. The multiplier phototube mounts on the scattering chamber and detects light forward scattered by any particles in the chamber. Further description of the photometer is given in 7.6.

5.9 *Flowmeter* (h)—A float-type flowmeter (rotameter) is used, capable of reading a flow rate well in excess of the maximum test rate to be used. A meter reading somewhat above 100 L/min is the common size. It must be protected against fouling by any DOP accumulation.

5.10 *Exhaust Pump or Blower* (i)—This can be either a positive displacement pump or blower or a multistage turbine-type blower. In any case, there must be more than sufficient capacity to draw air through the total resistance of test specimen, scattering chamber, flowmeter, and all of the related lines, valves, filters, etc., at the maximum test rate (usually 85 L/min).

5.11 *Cleanup Filter* (j)—This should be a filter of sufficiently high capacity and efficiency to remove smoke from the airstream before it passes through the flowmeter. Aerosol particles would ultimately affect the accuracy of the meter.

6. Procedure

6.1 It is necessary to have the equipment prepared and calibrated in advance of any test work. Once prepared and in adjustment, the equipment can be turned on at any time and operated as long as desired with only occasional minor readjustment. Instruction for preparation and operation of each item of equipment is given below.

6.2 *Aerosol Generator*—Turn on the air supply and heating units of the DOP aerosol generator. Wait until the aerosol output has been stabilized; usually this will require ½ h or more from a cold start. Draw a portion of the aerosol through the particle size analyzer, verify the aerosol particle size, and adjust generator conditions until particle diameter is 0.30 μm (by adjustment of quench air temperature).

6.3 *Adjustment of Photometer*—Using the same flow rate that will be used for the test specimen (usually 32 L/min ± 2 % through 100 cm^2 of area ± 2 % when testing filter media), aerosol from the generator is passed directly through the scattering chamber. Adjust the "Gain" potentiometer of the galvanometer circuit in the photometer until the meter reads 100.0.

6.3.1 Draw clean filtered air through the scattering chamber. Adjust for stray light so that the photometer meter reads zero on the most sensitive scale.

6.4 *Penetration Measurement*—Mount the sample to be tested in the sample holder, making certain that all seals are tight. Draw aerosol through the test specimen. Adjust flow rate to the desired level, for example, 32 L/min. Starting with the least sensitive range, use progressively higher sensitivity until a reading can be obtained.

6.4.1 Read the photometer. Report the value as percent DOP penetration.

6.5 *Flow Resistance*—At the beginning of the penetration test and after the desired flow rate has been established, the manometer is read, showing the resulting pressure drop across the test specimen. This may change after a short time due to accumulation of DOP in the test piece. Ordinarily, the reading is reported in millimetres of water pressure.

6.6 *Other Data*—Line temperature and manometer readings at the flowmeter should be noted along with the current barometer reading so that airflow rates may be reduced to standard conditions if this is required at some later time.

6.7 *Q Value*—When comparing the filter efficiency of various media, it is often useful to report filter performance in terms of the Q value. Calculate Q value as follows:

$$Q = [-100 \log (P/100)]/\Delta p$$

where:

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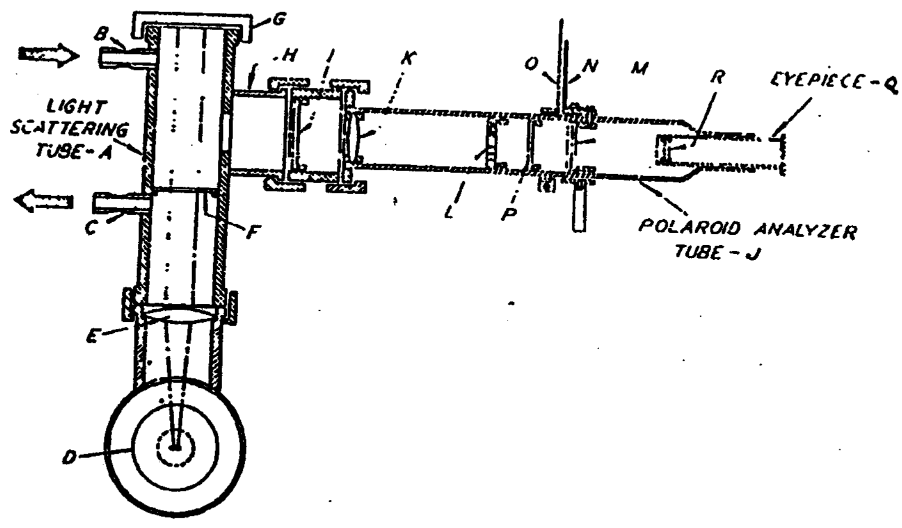

A—Smoke chamber and light-scattering tube
B—Smoke inlet port
C—Smoke outlet port
D—Light source
E—Lens to form parallel beam of light through smoke chamber
F—Baffle to reduce stray light scattered from walls of chamber
G—Velvet-lined cap on chamber
H—Side arm to light scattering tube
I—Smoke-retaining window
J—Polaroid analyzer tube
K—Light collecting lens
L—Bipartite disc (split field polaroid)
M—Polaroid disc mounted in movable collar
N—Indicator on polaroid holder to show angle of displacement from verticle axis
O—Degree scale for indicator (N)
P—Green filter for obtaining monochromatic light
Q—Adjustable eyepiece
R—Lens in eyepiece

FIG. 2 "Owl" Particle Size Meter

P = DOP penetration, %, and
Δp = airflow resistance, mm of water.

7. Discussion of Equipment

7.1 *Availability*—Although equipment for the DOP test is commercially available, some care must be exercised in its procurement. While the test is well developed and very reliable, the equipment is complex. It must be within certain design parameters and must be carefully fabricated. Design details are available[4] but it is strongly recommended that purchase be made of equipment from a professional fabricator.[7]

7.2 *Air Supply*—Any reliable air source may be used that is capable of supplying 120 L/min. If taken from a compressed air system, a stepdown regulator should be used, followed by a control value, and a good commercial air-line filter ahead of the high-efficiency (HEPA) filter.

7.2.1 For a mobile unit or where a compressed air line is not available, a blower can be used. A multistage blower, turbine-type, is effective and will run continuously with very little maintenance. An absolute (HEPA) type filter is used customarily in the supply line to ensure that the air is entirely clean.

7.2.2 In any case, provision must be made to either cool or heat the air as conditions may require. Commercial heat exchange equipment is satisfactory.

7.3 *Aerosol Generator*—The aerosol generator consists of a pot containing about 500 mL of DOP at about 170°C. DOP vapor is carried from the container by a small heated airstream (about 215°C) and mixed with a larger stream of quench air (about 25°C) to form the aerosol particles. The airstream temperatures determine particle size and, once established must be precisely controlled. The aerosol is generated continuously; excess aerosol is disposed of away from the test area.

7.4 *Owl*—A typical "Owl" is shown in Fig. 2. A reading of 29 ± 1° corresponds to 0.3-µm diameter DOP particles.

7.5 *Light-Scattering Chamber*—The light-scattering chamber shown diagrammatically in Fig. 3 is an essential component of the photometer system. Any airborne particles entering the chamber are strongly illuminated and the forward-scattered light intensity is detected by a multiplier phototube (H).

7.5.1 Light from the lamp bulb filament (A) is focused at the center of the light tube (E) by means of condensing lenses (B). An opaque patch (D) cemented to the inside facing of the condensing lens intercepts any direct light from the filament to the phototube.

7.5.2 The stop opening at (E) has a beveled edge, thus providing a knife edge periphery to the opening. This is important in keeping the amount of light scatter from this illuminated edge at a bare minimum.

7.5.3 Take every care to ensure that only light forward-scattered by the test aerosol is seen by the phototube. Following are points requiring special attention:

7.5.3.1 Optics must be accurately aligned with the optical axis. The light bulb filament image should be focused at the center of the light stop opening.

[7] Equipment suitable for the DOP test may be obtained from Air Technology, Inc., 1717 Whitehead Rd, Baltimore, MD 21207.

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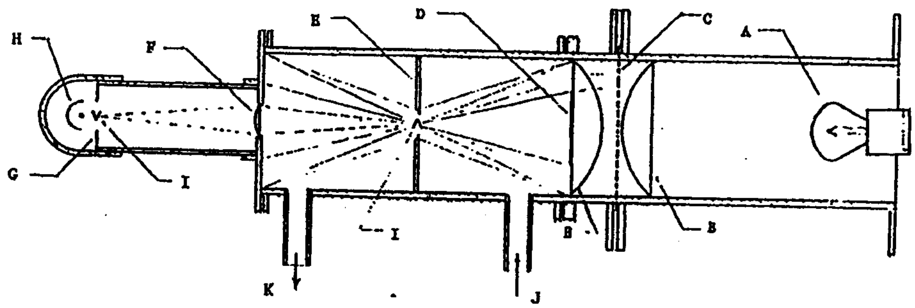

A—Light source
B—Front lenses
C—Optical filter or light screen
D—Opaque disk
E—Center light baffle
F—Back lens
G—Rear light baffle
H—Multiplier prototube
I—Filament image
J—Inlet port
K—Outlet port

FIG. 3 Light-Scattering Chamber—Schematic Section Showing Optical System

7.5.3.2 All lens surfaces must be perfectly clean; otherwise, light scatter will result.

7.5.3.3 All lens edges, especially if they are ground surfaces, should be painted with flat black.

7.5.3.4 No frosted or etched design or figure can be permitted on the light bulb envelop facing the condensing lenses.

7.5.3.5 The sharp edge of the light stop must be free of nicks, burrs, or even attached particles.

7.5.3.6 Light bulb current must be supplied from a voltage-stabilized circuit.

7.6 *Photometer*—Various types of photometers are in use. In most, a Type 931A multiplier phototube is operated at output currents below 30 μA to ensure stable operation. A scale switch permits the measurements down to 0.001 % or 0.0001 % penetration of DOP smoke.

8. Maintenance and Troubleshooting

8.1 When used routinely, the DOP test is reliable and seldom presents any serious problems. Experience will soon show the operator what steps must be followed to maintain fully satisfactory equipment performance. However, to assist the neophyte especially, the following basic suggestions are offered:

8.2 *Daily Care:*

8.2.1 Ascertain that the DOP liquid level or supply is adequate.

8.2.2 See that all temperatures are correct and that controls are functioning properly.

8.2.3 If supply air or quench air must be cooled, see that the cooling water is turned on.

8.2.4 Check aerosol particle size and make any adjustments needed to maintain 0.3-μm diameter.

8.3 *No Aerosol Generation:*

8.3.1 Make sure that air supply is working properly.

8.3.2 See that all heaters are on and working.

8.3.3 Check DOP level or supply.

8.3.4 See that both heated air and quench air are being supplied and at the proper rates.

8.4 *Particle Size Analyzer:*

8.4.1 See that the analyzer lamp is lighted.

8.4.2 Make sure that aerosol is flowing through the instrument (exhaust blower or pump turned on and the analyzer line valve open).

8.4.3 See that connection tubes to the analyzer are not pinched.

8.4.4 Adjust quench air temperature to attain correct particle size.

8.5 *Photometer Not Reading:*

8.5.1 See that the lamp is lighted in the scattering chamber.

8.5.2 See that the amplifier is plugged in and operating.

8.5.3 Make sure that aerosol is flowing through the scattering chamber (selector valve in "run" position and not "purge").

8.5.4 See that multipler phototube is mounted on the scattering chamber and that lead wires are connected in the amplifier.

8.6 *Photometer Cannot be Adjusted for 100 % Reading:*

8.6.1 Make sure that aerosol generator is operating properly.

8.6.2 Make sure that multiplier phototube is properly adjusted.

8.7 *Photometer Cannot be Adjusted for Stray Light:*

8.7.1 The usual fault is excessive stray light in the scattering chamber. Adjust position of the light source (instrument manual) to attain minimum reading on clean air.

8.7.2 Ascertain that the amplifier is performing correctly.

8.7.3 Have the phototube and connecting leads checked.

8.7.4 Make sure there are no leaks into the filtered air supply line. Leaks will introduce particulate matter and cause a meter reading.

8.7.5 If the problem persists, the source of stray light sometimes can be detected by removing the multiplier phototube and looking into the scattering chamber. This approach should be taken only by someone thoroughly familiar with the equipment.

9. Quality Control

9.1 Following are steps that can be taken to help ensure a reliable bias and agreement between laboratories:

9.1.1 *Calibrated Orifice Plate*—Orifice plates are available with calibrations traceable to the National Institute of Standards and Technology (formerly NBS). The plate is inserted in the sample holder and used as a flowmeter against

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which the instrument flowmeter is compared. For information regarding calibrated plates, inquire of the manufacturer.[7]

9.1.2 *Leaks* in the system can be serious since they will impair the accuracy of flow rate readings. Every precaution must be taken to ensure tightness in lines, instrument filters, and fittings.

9.1.3 *Reference Filter Paper*—It is convenient to have on hand a supply of high-efficiency filter paper that has been pretested and preferably cross-checked by at least one other laboratory. An occasional test run on one of these filter samples will be reassuring and indicative that all is well. Results may be plotted on a control chart to demonstrate attainment of statistical control of the measurement process.[8]

10. Keywords

10.1 atmospheric analysis; air assay media; collection efficiency evaluation of filters; dioctyl phthalate, use for filter evaluation; high efficiency filters; membrane filters; particle collecting devices; smoke test for filter evaluation

[8] Taylor, John K., "Quality Assurance of Chemical Measurements," Lewis Publishers, Inc., Chelsea, MI (1987).

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